

Accelerant classification by gas chromatography/mass spectrometry and multivariate pattern recognition

Beijing Tan^a, James K. Hardy^{a,*}, Ralph E. Snively^b

^a Department of Chemistry, The University of Akron, Akron, OH 44325-3601, USA

^b William Alvine Assoc., P.O. Box 1123, Marshalls Creek PA 18335, USA

Received 3 June 1999; received in revised form 6 March 2000; accepted 28 April 2000

Abstract

Petroleum-based accelerants are commonly associated with arson-related fire. Detection and correct classification of accelerant is crucial to arson investigation. The application of gas chromatography/mass spectrometry (GC/MS) and multivariate pattern recognition techniques for petroleum-based accelerant detection and classification is presented. The method feasibility and matrix effects on accelerant classification were studied using principal component analysis (PCA). A soft independent model classification analogy (SIMCA) model was then developed to evaluate evaporation, sample size, and sample charring. Depending on the sample class, the detection limits of correct classification were in the range of 5–50 μ l. The detection limits of closest classification could be as low as 1.25 μ l in the charred samples. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Fire debris; Accelerant; GC/MS; Pattern recognition; Principal component analysis; SIMCA

1. Introduction

Arson represents a serious problem both in the cost of lives and money. In 1994, the National Fire Protection Association estimated that the annual costs of fire-related damages are as high as \$6 billion, and up to 20% of all fires are caused by arsonists. Some 107,800 arson-related fires were reported and 550 people lost their lives [1]. In most cases involving arson, petroleum-based accelerants, such as gasoline, kerosene, and paint thinner, are used, because of their low cost and easy availability.

Petroleum-based accelerants are typically complex materials consisting of hundreds of components making their detection and identification difficult. The

situation is made worse by the fact that contamination is common as a result of the pyrolysis of materials such as plastics, carpet and carpet padding at the fire scene [2,3]. Gas chromatography/mass spectrometry (GC/MS) analysis of fire debris has been used to identify residues of petroleum-derived accelerants. Identification relies on two main types of pattern matching methods. One approach makes use of extracted ion profile matching [4,5]. With this method, intensity profiles for characteristic ions of fire debris samples are visually compared against the profiles of known petroleum distillates. Another method relies on target compound analysis [6,7]. A target compound chromatogram (TCC) is constructed using the retention time and relative amount for each target compound. Visual pattern recognition is employed to confirm the fit to TCCs of known accelerants for the identification of an unknown accelerant. Both

* Corresponding author. Tel.: +1-330-972-6061.
E-mail address: jkh@uakron.edu (J.K. Hardy).

methods require visual inspection for the identification of complex samples, which is time-consuming and may lead to misclassification.

Multivariate statistical techniques offer powerful tools in interpreting complex data. Principal component analysis (PCA) and soft independent model classification analogy (SIMCA) are two significant techniques for dealing with complex data interpretation. PCA relies on the linear transformation of the original set of measurements into a substantially smaller set of uncorrelated variables while retaining as much of information present in the original data set as possible. It gives a geometrical visualization of results in a lower dimensional space [8]. With SIMCA, PCA is first conducted on each sample class. The optimum number of PCs is determined by cross-validation and a confidence envelope is constructed to contain the data points. Sample data can then be projected onto each confidence envelope to determine to which category it belongs [9]. Category prediction of an unknown is based on Mahalanobis distances and the sample residue between the unknown and the SIMCA model. If both the residue and the Mahalanobis distance of the unknown are within their threshold after the unknown is projected on the model of a class, this unknown is assigned to this class. PCA and SIMCA have been successfully applied to interpret complex data in a number of studying areas, such as design and characterization of chromatographic systems [10,11], sensors [12,13], environmental chemistry [14,15], food [16,17] and other industrial applications [18–20].

In this study, we report the application of PCA and SIMCA for accelerant identification and classification. The results demonstrated that these techniques could be successfully used to classify accelerants.

2. Experimental

2.1. Materials

Methylene chloride (GC Resolv grade, Fisher Scientific) was used as the solvent for extracting accelerants from the listed matrices. *d*₈-Naphthalene (Sigma, >99%) was used as an internal standard. Accelerants numbering 51 in five classes were evaluated. Based on the ASTM Flammable and Combustible Liquid Classification System [21], all samples were initially classified and are listed in Table 1. Three types of wood:

Table 1
Samples and their manufacturers

Sample name	Brand
Class 1	
Lacquer thinner	Recochem Inc.
Turbo octane boost	N. American Oil
Engine starting fluid	IG-LO Inc.
75725 Texsolve B	Texaco
75726 Texsolve C	Texaco
Lacquer thinner	Parks
Class 2	
Zippo lighter fluid	Zippo
Jone-E fluid	Orbex Inc.
Kensol-80	Kendall
Kensol-10	Kendall
75729 Texsolve V	Texaco
75724 Skellite	Texaco
Gasoline, regular	BP oil
Gasoline, plus	BP oil
Gasoline, premium	BP oil
Gasoline, premium	Shell oil
Gasoline, regular	Shell oil
Gasoline, plus	Shell oil
Klenk's thinner	Zynolyte
Class 3	
75740 Texsolve s/LO	Texaco
Thompson's waterproofer	Thompson & Formby
Marvel Mystery oil	Marvel oil company
Parks mineral spirit	Parks
STP lead substitute	STP
Kensol-30	Kendall
75731 Texsolve S	Texaco
Kerosene	ARCO
Steam distilled turpentine	Parks
Hearth and grill charcoal starter	Tradco Corporation
Kingsford odorless charcoal fluid	Kingsford Products
Nasco mineral spirit	National Solvent
House furniture cleaner	Holloway House Inc.
Class 4	
Kerosene	BP oil
Gumout xtra fuel injector cleaner	Pennzoil
STP superconcentrated fuel injector cleaner	STP
STP superconcentrated valve cleaner	STP
STP gas treatment	STP
STP diesel fuel treatment & injector cleaner	STP
Jet fuel	Ira Price
Kensol-48T	Kendall
Kensol 50T	Kendall
Kerosene	Parks
Avjet A	Texaco
STP fuel injector and carb cleaner	STP
STP lead substitute with octane booster	STP

Table 1 (Continued)

Sample name	Brand
Class 5	
No. 2 diesel	BP oil
Diesel supreme	BP oil
No. 2 fuel oil	Kendall
Kensol-51	Kendall
Kensol-61	Kendall
Kensol-53	Kendall

white pine, oak, and poplar and three kinds of carpet with different piles: polyolefin (Royal Scot), nylon (Queen Carpet Company), and polypropylene (Shaw Industrial Inc.) were used as material matrices. Samples and material matrices were obtained through the courtesy of various chemical manufacturers, or purchased locally.

2.2. Methods

2.2.1. GC/MS analysis

Samples were assayed using a Hewlett-Packard (HP) 5890 gas chromatograph (GC) with an HP 5970 series mass selective detector (MSD). The GC was equipped with an HP 7673A automatic sampler. Automation and data acquisition were handled by an MS ChemStation (HP UNIX series). A 25 m \times 0.2 mm \times 0.5 μ m capillary column, with a stationary phase of 5% cross-linked methyl silicone (Supleco), was used. For analysis, a single temperature ramp was used. The initial temperature was 35°C which was held for 3 min. The temperature was increased from 35 to 270°C at the rate of 12°C/min, and held at 270°C for 7 min. The total run time was 30 min. The injection volume was 1.0 μ l and the total flow of carrier gas was 40 ml/min helium (ultra-high purity, PRAXAIR Inc.). To increase sensitivity and minimize the interference of pyrolysis products, selective ion mode (SIM) of mass data acquisition was employed with the following lines: 55, 57, 69, 71, 91, 105, and 141. These mass lines were selected after evaluation of the initial full scan of each sample with GC/MS. In addition, the fragments with these mass numbers are the major daughter ions or molecular ions of the accelerants. The data collected with these lines produces the same PCA result as the data collected with scan mode.

2.2.2. Initial training set preparation

A volume of 1 ml of the internal standard (0.40 g *d*₈-naphthalene in 100 ml methylene chloride) and 100 μ l accelerant were added into a 25 ml volumetric flask, which was then filled to the mark with methylene chloride.

2.2.3. Matrix exposed accelerant sample preparation

A quantity of 3 g of a matrix was placed in a 100 ml-glass bottle to which 100 μ l of an accelerant was added. The bottle was capped for 24 h at 20°C. The accelerant was then extracted using 20 ml of methylene chloride and then diluted to 25.0 ml after addition of the internal standard. All samples were prepared in triplicate. A sander was employed for making sawdust of the wood matrix.

2.2.4. Effect of evaporation

Evaporation effects were evaluated using a 40 l tank with an aluminum board as cover. The temperature in the tank was regulated by placing it in a water bath. Relative humidity was maintained at 50% by blending a humid and dry air stream and introducing it at a rate of 500 ml/min. Samples were placed on individual watchglasses within this tank. A 100 μ l representative accelerant from each class was exposed to polyolefin-piled carpet at temperature of 0, 20, and 35°C for up to 7 days. Samples were extracted and analyzed as in the matrix effect study.

3. Data processing

After GC/MS analysis, the resulting data files required initial preprocessing for the creation of a data matrix suitable for subsequent data analysis. A computer program, written in-house to run on a Sun SparcStation, was used for this purpose. With this program, a response versus time profile was produced by extracting the GC/MS data file of each accelerant sample. In the resulting data matrix, the values in each row represent the measurements of different variables for each sample, and the values in each column represent the measurements of different samples for a variable. Each replicate was treated as an individual sample in the data matrix.

For each GC/MS data file, the range of 60–1029 s was divided into 19 equal parts and the signal in each

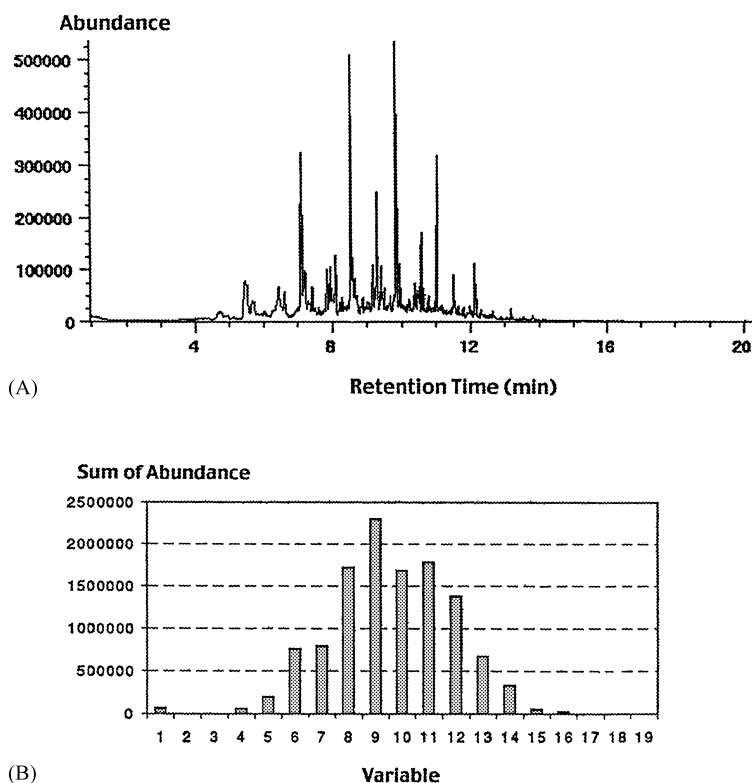


Fig. 1. Typical GC/MS chromatogram of an accelerant (A) and its profile of response vs. time (B). Accelerant: jet fuel.

part was summed to represent a variable in the data matrix. Fig. 1 shows the chromatogram of an accelerant and its corresponding bar graph of the generated data set. For each chromatographic data, the response was normalized to the area of the internal standard to account for run-to-run variations. In addition, based on the retention time of the internal standard, the data matrix was time-normalized to account for variations in column performance. The initial data process was conducted on Sun SparcStation. PCA and SIMCA were carried out with MATLAB 4.2 (The MathWorks Inc.) and PLS Toolbox (Eigenvector Technologies Inc.) on PowerMac.

4. Results and discussion

4.1. Principal component analysis

PCA was first conducted on the initial training set, which was preprocessed from GC/MS data of the

dilute petroleum products, to assess the feasibility of PCA application in petroleum classification. By performing PCA on the initial training set, a series of principal components (PCs) could be obtained. Each PC is associated with an eigenvalue. Principal component 1 (PC1) has the largest eigenvalue and carries the largest variance of the original data, and subsequent PCs carry variance in a decreasing order. The first two PCs account for about 57.1% of the total variance in the original data set. Fig. 2 shows the score plot of PC1 versus PC2. In the score plot, each class tends to cluster together and is well differentiated from other classes.

PCA was employed to investigate the effect of matrix material to which the petroleum products may be applied in an arson fire. Each matrix was extracted with methylene chloride and the extract was analyzed with GC/MS. It was found that only in the case of white pine and poplar were some additional peaks observed. However, they evolved after 20 min, presenting

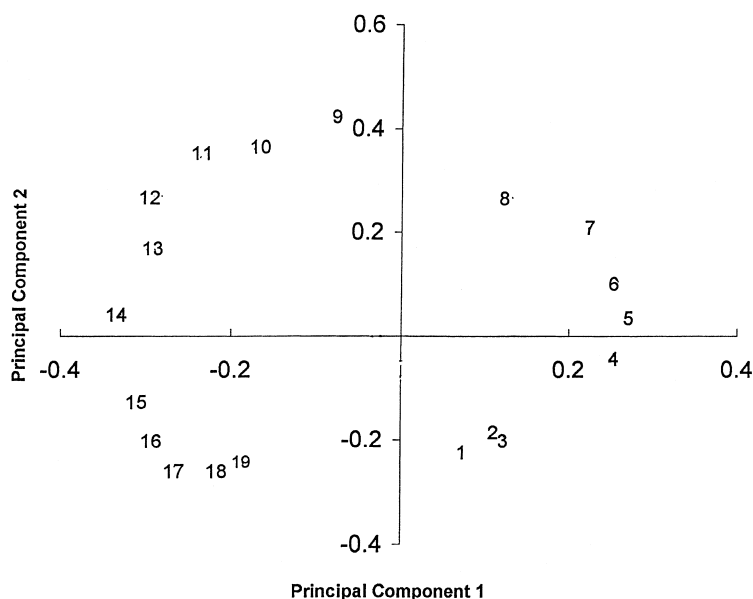


Fig. 2. Loading plot of PC1 and PC2 for dilute accelerant data set.

no problem with the procedure. A series of samples were then prepared by exposing 3.0 g of each matrix to 100 μ l of gasoline. Samples were allowed to stand for 24 h at room temperature in sealed glass bottles and were subsequently extracted with methylene chloride. The PC1 versus PC2 score plot of these samples is given in Fig. 3. It is evident that residue collected from carpet provides results that are similar to the neat samples. Compared to accelerants applied to carpet, samples applied to wood dust deviate significantly from the original samples.

The whole training set was re-evaluated by placing accelerants on polyolefin-piled carpet since it was found to be a better source for collecting accelerant residue, compared to the woods investigated. Fig. 4 shows the score plot of PC1 and PC2 for the accelerants weathered with polyolefin-piled carpet. It shows that the score plots of dilute samples and matrix-exposed samples present near-identical results.

4.2. SIMCA

SIMCA, a supervised learning technique, was used to create a PCA model for each class with which unknowns could be predicted. Among the triplicate matrix-exposed samples, a SIMCA model was created

using two of the replicates. The optimum number of PCs used for each class was determined by cross-validation. The third replicate for each sample was tested with the resulting model. A PCA model for each class was constructed with a different number of optimum PCs. Two, three, six, four, and two PCs were used to construct class 1–5 PCA models with the captured variance of 71, 92, 96, 94, and 94%, respectively. All training data and test data were correctly classified with this SIMCA model. The remaining studies were done via this SIMCA model to establish criteria for sampling and correct classification.

4.2.1. Factors that could alter classification

After ignitable liquid was detected in a sample of fire debris, there are four possible SIMCA prediction results: correctly classified to its original class, closely classified to its original class, closely classified to another class, and classified as being in another class. Both evaporation and accelerant quantity could affect successful classification. As the accelerant amount decreases, a point can be reached where some components are undetectable. Consequently, the pattern may change and a sample could either be misclassified or not classified. In addition, evaporation would also affect accelerant classification. The fact

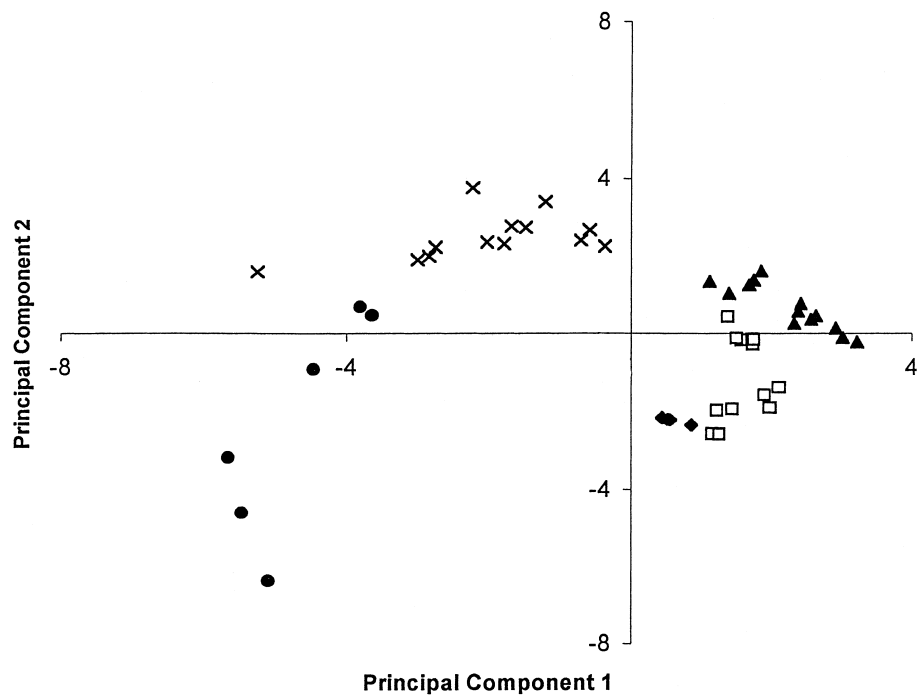


Fig. 3. Score plot of PC1 and PC2 for dilute accelerants: (◆) class 1; (□) class 2; (▲) class 3; (×) class 4; (●) class 5.

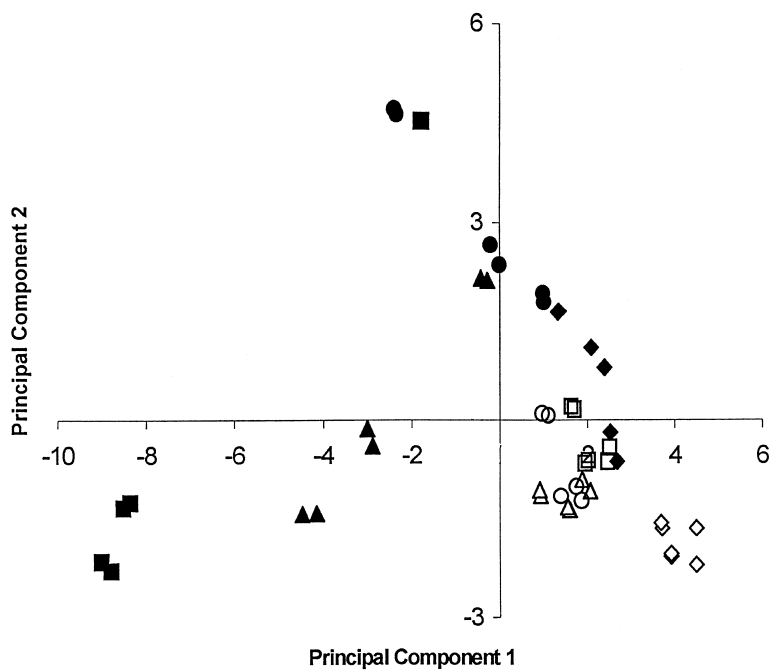


Fig. 4. Score plot of PC1 and PC2 for the data set of gasoline exposed to different matrices: (◇) unexposed; (◆) air; (Δ) nylon carpet; (□) polyolefin carpet; (○) polypropylene carpet; (●) white pine; (▲) oak; (■) poplar.

Table 2
Petroleum distillate classification system

Class number	Class name	<i>n</i> -Alkane peak spread	Example
1	Light petroleum distillate	C ₄ –C ₁₁	Petroleum ethers, pocket lighter fuel, some rubber cement solvents, skelly solvents, M&P naphtha, some camping fuels
2	Gasoline	C ₄ –C ₁₂	All brands and grades of automotive gasoline, including gasohol
3	Medium petroleum distillates	C ₈ –C ₁₂	Painter thinners, mineral spirits, some charcoal starters, some torch fuels, some paint thinner, some solvents for insecticides and polishes, some land oil
4	Kerosene	C ₉ –C ₁₇	No. 1 fuel oil, Jet-A fuel, insect sprays, some charcoal starters, torch fuels
5	Heavy petroleum distillates	C ₉ –C ₂₃	No. 2 fuel oil, diesel fuel

that the components of an accelerant evaporate unevenly changes the pattern of accelerant sample and leads to misclassification. Accelerant evaporation is mainly controlled by temperature and exposure time. The time limit for correct classification of each class accelerant was determined using the SIMCA model after known evaporation time. The time limit of accurate detection and classification was also determined for each class accelerant.

4.2.2. Accelerant quantity

To determine the effect of accelerant amount, accelerant volumes of 5–75 μ l (4–60 mg) were added to 1 in.² samples of polyolefin-piled carpet (about 3 g). Each sample was then stored in a capped 100 ml glass bottle at 20°C for 24 h. All were then extracted and analyzed as in the matrix effect study. The results are presented in Table 2. Accelerant of class 1 exhibits a relatively small number of components and the peak distribution is very narrow. As a result, samples of this class were correctly classified with amounts as low as 5 μ l. Although accelerant of class 5 has a broad peak distribution, correct classification could be made at the same level. The reasons are that class 5 has the

highest boiling point range and no other class could be formed beyond it. Accelerants of classes 2–4 have relatively high correct classification limits, which are 1.0, 2.0, and 2.0 nl, respectively. For these classes, accelerants overlap with those of adjacent class and low abundance components in an accelerant may not be detected at a low concentration, which leads to pattern change and misclassification.

4.2.3. Temperature and exposure time

The data resulting from evaporation at different temperatures and various lengths of time were predicted with the SIMCA model. The amount of accelerant retained in the carpet was calculated. The results are shown in Table 3. Temperature and exposure time have a combined effect on accelerant evaporation. At any temperature, more accelerant will evaporate as exposing time increases. The same is true at any time as temperature is increased. Table 3 shows that, for each class, the time limit for correct classification decreases as the exposure temperature increases. For example, the time limit for correct classification of class 3 accelerant decreases from 12 to 3 h as temperature increases from 0 to 35°C. The temperature effect on

Table 3
Variable reduction

	New variable				
	1	2	3	4	5
Original variable	1–2	3–5	6–8	9–14	15–19
Retention time (s)	60–162	162–315	315–468	468–774	774–1029
Peak spread of <i>n</i> -alkane	C ₆ –C ₈	C ₈ –C ₁₀	C ₁₀ –C ₁₂	C ₁₂ –C ₁₇	C ₁₇ –C ₂₂
ASTM range	C ₄ –C ₁₁	C ₄ –C ₁₂	C ₈ –C ₁₂	C ₉ –C ₁₇	C ₉ –C ₂₃

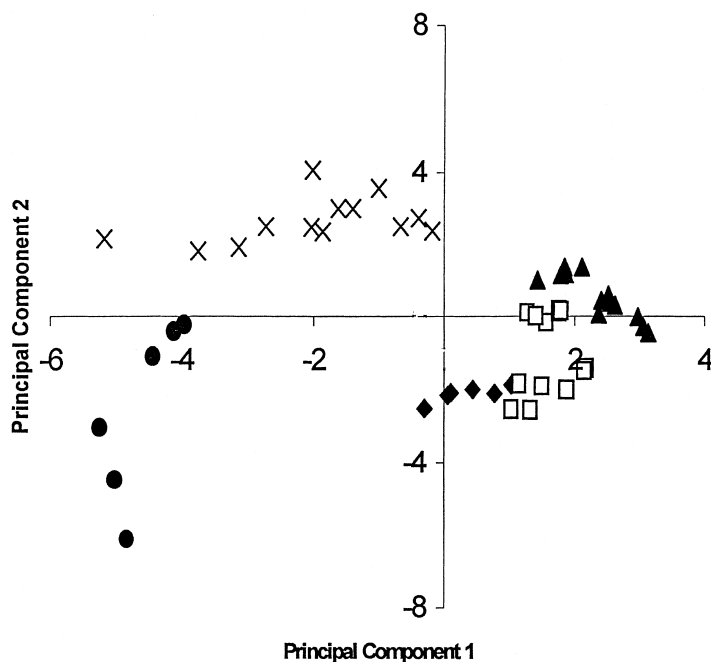


Fig. 5. Score plot of PC1 and PC2 for the data set of matrix-exposed accelerants: (◆) class 1; (□) class 2; (▲) class 3; (×) class 4; (●) class 5.

accelerant evaporation could be observed in terms of the accelerant amount retained after evaporation. Only 37% of the class 2 accelerant remained after 4 h at 0°C, while only 25% remained at 35°C after 45 min. The time limit for close classification and the accelerant amount retained in the carpet are also presented in Table 3. The time limit for close classification has the same trend as the time limit for correct classification. Beyond the time limit for close classification, no accelerant could be detected.

4.2.4. Matrix charring

Polyolefin-piled carpet was decomposed in a pyrolysis oven and the product was collected in iced methylene chloride (−10°C). GC/MS analysis of the obtained solution is shown in Fig. 5. As can be seen, there are many components in the pyrolyzate of polyolefin-piled carpet. The peaks of these components will obscure an accelerant pattern and make accelerant identification impossible. However, GC/MS analysis of polyolefin-piled carpet charred in an open air shows no peak in the same range. The reason is that, in an open air, com-

bustion and evaporation of carpet burning products occur.

4.3. SIMCA model validation

When the SIMCA model was developed, it was validated by cross-validation (leave-one-out validation). A confidence envelope for each class was derived by carrying out a PCA separately for each class. The number of PCs used to construct each confidence envelope was optimized by leave-one-out validation. The final

Table 4
SIMCA model and prediction

Class	No. of training samples	No. of PCs	Variance captured (%)	Correct classification
1	24	2	71.35	12/12
2	52	3	91.96	26/26
3	52	6	96.03	26/26
4	52	4	94.48	26/26
5	12	2	93.87	12/12

Table 5
Concentration limit for correct classification by SIMCA^a

Accelerant volume/class (μl)	1	2	3	4	5
75	C ^a	C	C	C	C
50	C	C	C	C	C
25	C	C	M	M	C
10	C	M	M	M	C
5	C	M	M	M	C
Limit of correct classification (nl)	0.2	1.0	2.0	2.0	0.2

^a C: correct classification; M: closest classification.

SIMCA model not only correctly classified the training data set but also correctly predicted the test data set.

The SIMCA model was further validated using simulated fire debris. In an intentionally set fire, accelerant

is usually poured on the flooring carpet and the carpet is saturated with accelerant. In this study, this process was simulated by applying 1.0 ml accelerant on a 1 in.² polyolefin-piled carpet (about 3 g) and igniting the carpet, allowing the pile to combust. The fire was then extinguished. A representative accelerant of each class was tested in triplicate. The residue was extracted and analyzed with GC/MS. The results are presented in Table 4. It is shown that all representative accelerants could be correctly classified with the SIMCA model built previously. The recovery of accelerants is also presented in Tables 4–7. After being charred with polyolefin-piled carpet, accelerants of class 1 have the least recovery (2.5–14%), while accelerants of class 5 have the largest recovery (45–65%) because of their different volatility.

Table 6
Effect of temperature and exposure time on accelerant classification

Class	0°C		20°C		35°C	
	Correct (h (%))	Close (h (%))	Correct (h (%))	Close (h (%))	Correct (h (%))	Close (h (%))
1	3 (5.25)	3.25 (2.33)	0.5 (17.45)	0.75 (2.56)	0.4 (7.95)	0.5 (2.55)
2	4 (36.99)	10 (1.63)	1 (56.85)	3 (1.15)	0.75 (25.37)	1.25 (1.26)
3	12 (71.88)	72 (10.18)	6 (75.84)	48 (17.84)	3 (78.26)	24 (20.40)
4	>168 (37.82)		144 (33.45)	>168 (26.28)	24 (73.29)	96 (9.58)
5	>168 (40.24)		>168 (34.09)		168 (11.92)	

Table 7
Recovery of accelerants charred with carpet and their prediction with SIMCA model

Class	Retention time (min)	Trial 1 (%)	Average (%)	Trial 2 (%)	Average (%)	Trial 3 (%)	Average (%)	Correct prediction
1	1.467	2.52	2.52	13.58	13.58	13.19	13.19	6/6
2	5.035	19.85	20.43	35.70	36.97	38.17	40.25	6/6
	5.609	20.96		36.17		39.63		
	7.254	20.48		39.05		42.94		
3	5.694	35.77	38.51	51.61	51.71	49.72	49.48	6/6
	7.258	40.16		51.00		48.74		
	7.889	39.55		53.27		50.96		
	8.335	38.56		50.97		48.52		
4	7.247	39.30	42.37	33.94	42.18	32.37	42.40	6/6
	9.981	38.09		42.94		33.90		
	10.06	40.72		47.73		44.76		
	10.681	46.73		40.37		42.02		
	12.209	47.01		45.89		47.96		
5	7.178	40.81	45.34	64.25	64.98	59.21	59.38	6/6
	9.96	41.32		66.62		62.09		
	12.285	45.24		63.51		57.77		
	13.331	50.21		68.00		60.73		
	15.253	49.13		62.54		57.09		

5. Conclusion

It has been demonstrated that pattern recognition techniques (PCA and SIMCA) could be successfully applied to classify accelerants after GC/MS analysis. Two types of matrices were evaluated and carpet was found to be better than wood for sample collection. To identify the accelerant of any class correctly, the sample should be collected with 30 min after accelerant was applied at the temperature of 20°C. However, gasoline, a class 2 material, is often used in arson fire. Samples collected within 3 h can still be properly classified via the SIMCA model at 20°C. The correct classification limit is 0.8 µg for class 2 and 5 samples, 1.6 µg for class 3 and 4 accelerants, and 0.16 µg for class 1 accelerants. These values can be reduced if larger samples are obtained.

References

- [1] Annual Arson Report, US National Fire Protection Association, Quincy, MA, 1994.
- [2] W.J. Bertsch, *Chromatography A* 674 (1994) 329.
- [3] D.J. Dehaan, K. Bonarius, *J. Forensic Sci. Soc.* 28 (1988) 299.
- [4] A.J. Vella, *J. Forensic Sci.* 32 (1992) 131.
- [5] A.P. Koussiafes, W. Bertsch, *J. Chromatogr. Sci.* 3 (1993) 137.
- [6] R. Keto, *J. Forensic Sci.* 40 (1995) 412.
- [7] C.J. Lennard, V.T. Rochaix, P. Margot, K. Huber, *Sci. Justice* 35 (1995) 19.
- [8] Z. Juvancz, T. Cserhati, K.E. Markides, J.S. Bradshaw, M.L. Lee, *Chromatographia* 38 (1994) 227.
- [9] M.A. Sharaf, D.L. Illman, B.R. Kowalski, in: P.J. Pelving, J.D. Winefordne (Eds.), *Chemometrics*, Wiley, New York, 1986.
- [10] R.L. Deisenhofer, K. Ballschmiter, *Fresenius' J. Anal. Chem.* 36 (1998) 763.
- [11] R.J.M. Vervoort, M.W.J. Derksen, A.J.J. Debets, *J. Chromatogr. A* 765 (1997) 157.
- [12] F. Winqvist, P. Wide, I. Lundstrom, *Anal. Chim. Acta* 357 (1997) 21.
- [13] J. Getino, M.C. Horrillo, J. Gutierrez, L. Ares, J.I. Robla, C. Garcia, *Sens. Actuators B* B43 (1997) 200.
- [14] J. Mendez, A.J. Quejido, R. Perez-Pastor, M. Perez-Garcia, *Anal. Chim. Acta* 283 (1993) 528.
- [15] T.A. Delvalls, J.M. Forja, E. Gonzalez-Mazo, A. Gomez-Parra, *Trends Anal. Chem.* 17 (1998) 181.
- [16] L.M. Headley, J.K. Hardy, *J. Food Sci.* 54 (1989) 1351.
- [17] I. Moret, G. Scarponi, P. Cescon, *Agric. Food Chem.* 42 (1994) 1143.
- [18] H. Wilken, H. Schulten, *Fresenius' J. Anal. Chem.* 355 (1996) 157.
- [19] C.A. Drumm, M.D. Morris, *Appl. Spectrosc.* 49 (1995) 131.
- [20] J.F. Aust, M.K. Higgins, P. Groner, S.L. Morgan, M.L. Myrick, *Anal. Chim. Acta* 293 (1994) 119.
- [21] ASTM Designation: E 1618-94.